

$$\begin{array}{c}
 \text{H}_2\text{N}-\left(\text{CH}_2\right)_n-\text{CH}(\text{NH}-\text{Boc})-\text{COOH} \\
 n = 1 \text{ to } 3
 \end{array}
 \xrightarrow[\text{THF/water, RT, 18 h}]{\text{ROSu, K}_2\text{CO}_3}
 \begin{array}{c}
 \text{R}-\text{NH}-\left(\text{CH}_2\right)_n-\text{CH}(\text{NH}-\text{Boc})-\text{COOH} \\
 n = 1 \text{ to } 3
 \end{array}
 \xrightarrow[\text{THF, RT, 20 h}]{\text{NHS, DCC}}
 \begin{array}{c}
 \text{R}-\text{NH}-\left(\text{CH}_2\right)_n-\text{CH}(\text{NH}-\text{Boc})-\text{COOSu} \\
 n = 1 \text{ to } 3
 \end{array}
 \xrightarrow[\text{THF/water, RT, 18 h}]{\text{H}_2\text{N-spacer-NH}_2, \text{K}_2\text{CO}_3}
 \begin{array}{c}
 \text{R}-\text{NH}-\left(\text{CH}_2\right)_n-\text{CH}(\text{NH}-\text{Boc})-\text{X}-\text{NH}-\left(\text{CH}_2\right)_m-\text{CH}(\text{NH}-\text{Boc})-\text{COOH} \\
 31 - 92 \%
 \end{array}$$

R = hydrocarboxyl chain as defined

$$\begin{array}{c}
 \text{R}-\text{NH}-\left(\text{CH}_2\right)_n-\text{CH}(\text{NH}-\text{Boc})-\text{COOH} \\
 n = 1 \text{ to } 3
 \end{array}
 \xrightarrow[\text{RT, 1 - 2 h}]{\text{HCl}_{\text{conc}}, \text{MeOH}}
 \begin{array}{c}
 \text{R}-\text{NH}-\left(\text{CH}_2\right)_n-\text{CH}(\text{NH}-\text{Boc})-\text{COOH} \cdot \text{HCl} \\
 81 - 95 \%
 \end{array}$$

$$\begin{array}{c}
 \text{R}-\text{NH}-\left(\text{CH}_2\right)_n-\text{CH}(\text{NH}-\text{Boc})-\text{COOH} \\
 n = 1 \text{ to } 3
 \end{array}
 \xrightarrow[\text{CH}_2\text{Cl}_2, \text{RT, 1 h}]{\text{TFA}}
 \begin{array}{c}
 \text{R}-\text{NH}-\left(\text{CH}_2\right)_n-\text{CH}(\text{NH}-\text{Boc})-\text{COOH} \\
 90 \%
 \end{array}$$

$$\begin{array}{c}
 \text{R}-\text{NH}-\left(\text{CH}_2\right)_n-\text{CH}(\text{NH}-\text{Boc})-\text{COOH} \\
 n = 1 \text{ to } 3
 \end{array}
 \xrightarrow[\text{THF/water, RT, 16 h}]{\text{K}_2\text{CO}_3, \text{peptide-OSu}}
 \begin{array}{c}
 \text{R}-\text{NH}-\left(\text{CH}_2\right)_n-\text{CH}(\text{NH}-\text{Boc})-\text{CO-peptide} \\
 30 - 92 \%
 \end{array}$$

$$\begin{array}{c}
 \text{R}-\text{NH}-\left(\text{CH}_2\right)_n-\text{CH}(\text{NH}-\text{Boc})-\text{CO-peptide} \\
 n = 1 \text{ to } 3
 \end{array}
 \xrightarrow[\text{RT, 1 - 2 h}]{\text{HCl}_{\text{conc}}, \text{MeOH}}
 \begin{array}{c}
 \text{R}-\text{NH}-\left(\text{CH}_2\right)_n-\text{CH}(\text{NH}-\text{Boc})-\text{CO-peptide} \cdot \text{HCl} \\
 78 - 95 \%
 \end{array}$$

X:

$$\begin{array}{l}
 \text{---}(\text{CH}_2)_p\text{---} \\
 \text{---}(\text{CH}_2)_p\text{---N=N---}(\text{CH}_2)_p\text{---} \\
 \text{---}(\text{CH}_2)_p\text{---N---}(\text{CH}_2)_q\text{---N---}(\text{CH}_2)_p\text{---}
 \end{array}$$
 p = 1 to 10
 p = 1 to 4
 p = 1 to 4, q = 1 to 4

Figure 2

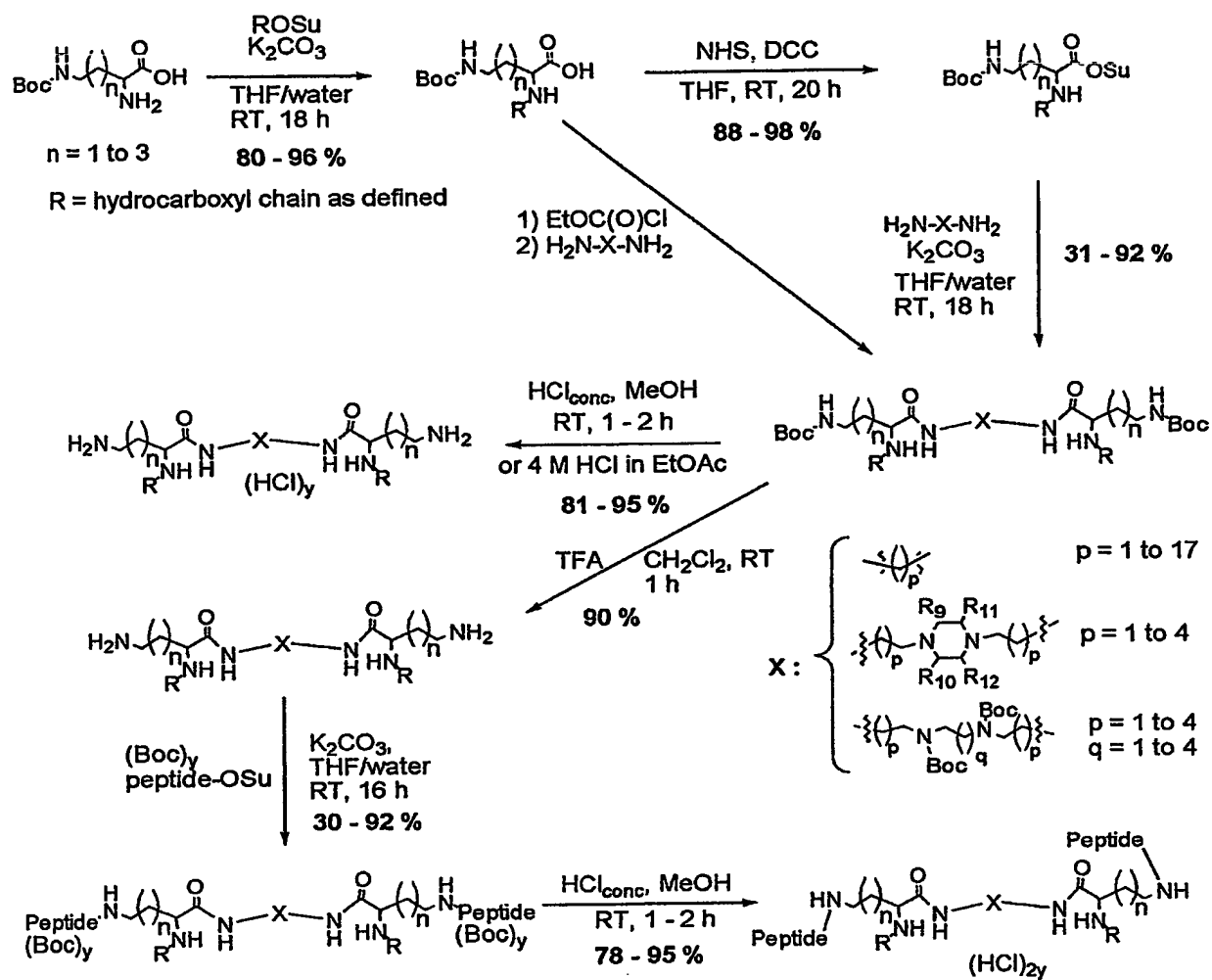


Figure 3

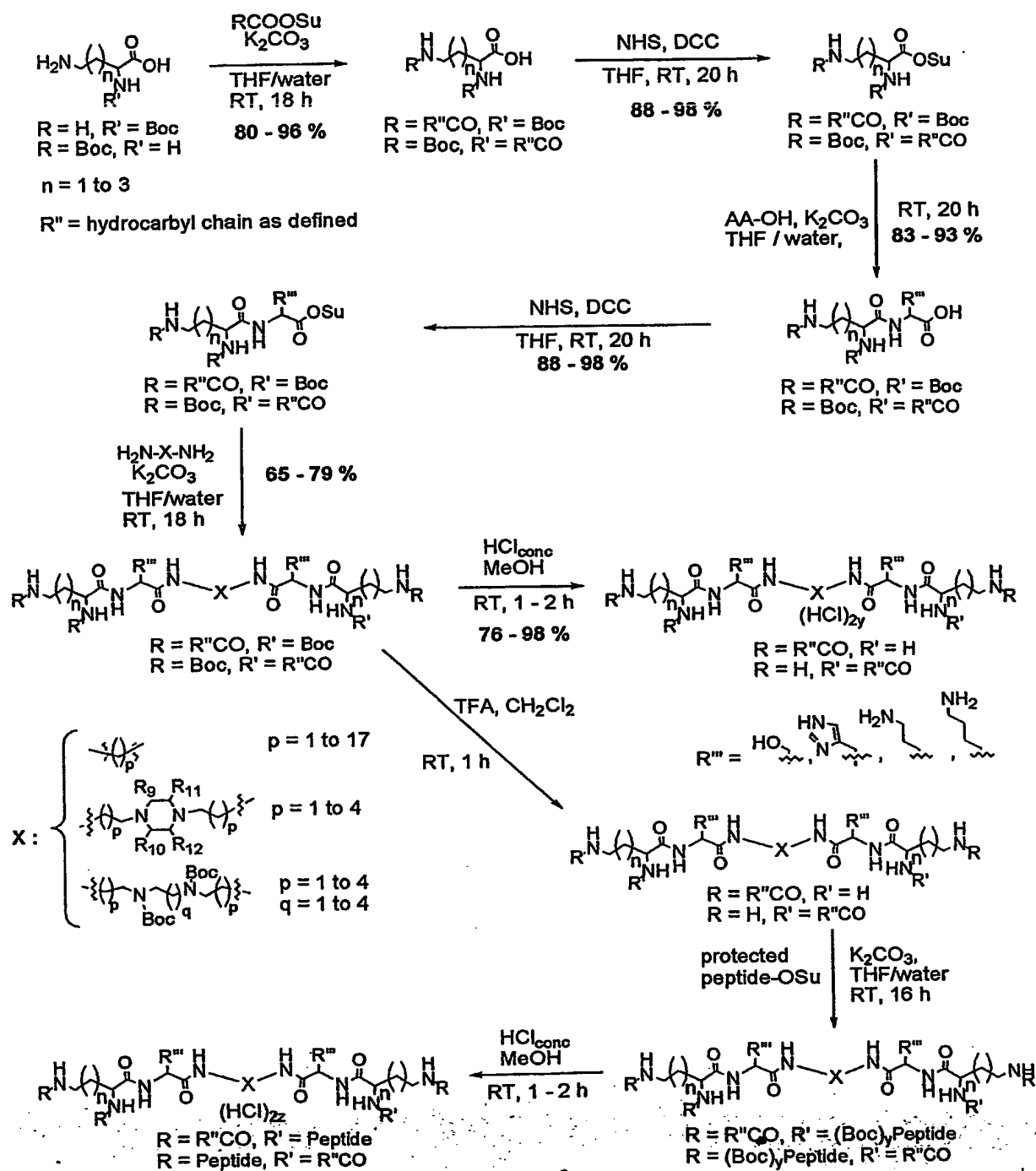


Figure 4

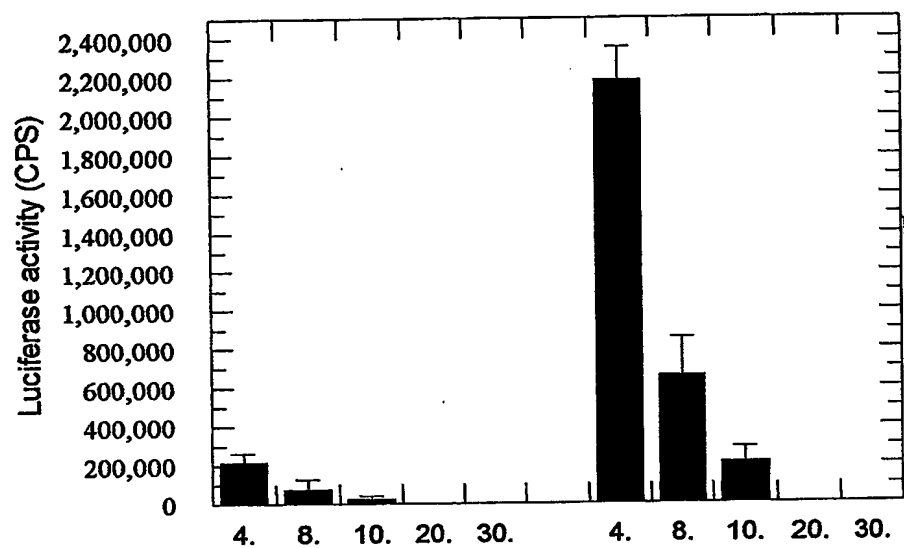


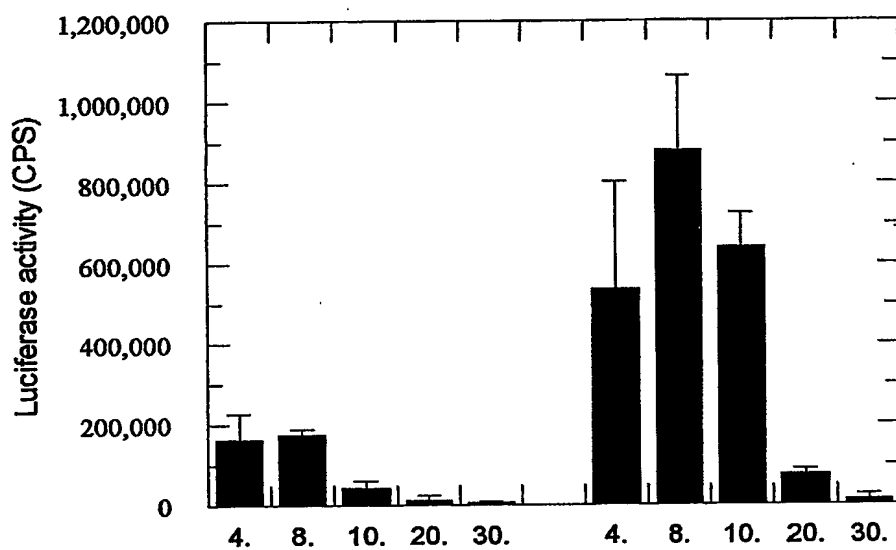
Figure 5

Figure 6

